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SULVANITE FROM UTAH

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INTRODUCTION

The rare copper vanadium sulphide, sulvanite, was found close to the surface, near the old mining camp of Mercur, Utah, in a fissure in carboniferous limestone, which is underlain by quartzite and overlain by red shale. This is the second authentic occurrence and the fourth reported one for this rare mineral. Sulvanite was first described from Australia and the two doubtful localities are in Argentina and in Siberia.

Most of the sulvanite from Utah occurs as cleavable masses; a few small crystals were found, however, whose measurements showed that the mineral is isometric. This crystallization had previously been suggested by several writers, on the basis of optical examinations by reflected polarized light on polished surfaces of material from Australia.

OCCURRENCE

The specimen from Utah is a breccia of fine-grained black limestone in a matrix of coarsely crystallized white calcite which also cuts the fragments of black limestone as small veinlets. The sulvanite is embedded in the coarse white calcite and also lies at its contact with the black limestone. Aggregates of small crystals of quartz are present in the white calcite.

The sulvanite occurs massive and as rough cubical crystals, somewhat elongated in one direction. The largest exposed cubical cleavage surface of the sulvanite extends for nearly a centimeter and a half. Small crystals are also developed in minor fractures in the white calcite and were isolated by treatment with dilute acid and measured on the goniometer.

PROPERTIES

The color of the larger masses of sulvanite is gray, resembling that of certain types of tetrahedrite. Some surfaces have a distinct bronzy appearance, in part iridescent and in part coated with secondary greenish minerals, which also stain the white calcite in places. The secondary minerals are chiefly green malachite and other green and greenish-yellow earthy alteration products. The yellowish color of some of this material suggests the presence of vanadium.

All the large cleavage surfaces of a single mass of sulvanite show two additional rectangular cleavages, so that there are three cleavages present, all mutually perpendicular. The cleavage is cubic, some of the crystals breaking readily into parallel slabs. All the rectangular traces of the cubic cleavage are prominent on the larger cleavage surfaces of the mineral in the matrix, but on some of the small crystals measured the cleavage traces are developed in only one direction.

The three rectangular cleavages have been referred to three pinacoids by Dieseldorf[1] and by Frebold[2] who, however, states that no polarization effects were observed, agreeing with the determinations of Orcel, de Jong, and Short[3] that optically the mineral is isotropic.

The interpretation of the three rectangular cleavages as cubic (isometric) is more in agreement with the other results obtained. The photomicrographs of sulvanite from Australia, shown by Frebold (p. 325) and by de Jong (p. 524) are identical in appearance in "white color," rectangular cleavages, and occurrence of alteration products with that observed on the sulvanite from Utah.

CRYSTALLOGRAPHY

The small crystals, isolated from the fractures in the white calcite, are about a millimeter thick and are somewhat unequal in length in different directions, so that they may be designated as slightly flattened and slightly elongated cubes. The surfaces have been somewhat dulled by the acid used in dissolving the calcite matrix but the measurements suffice to show that the crystals are isometric.

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[1] Dieseldorf, A., Sulvanite, ein primäres Vanadium-mineral: *Zeit. f. prakt Geol.*, **1901**, p. 421. *Zeit. d. Deutsch. Geol. Gesellsch.*, vol. **53**, Mon.-Ber. pp. 43–44, 1901.

[2] Frebold, G., Ueber einige Mineralien der Enargitgruppe und ihre paragenetischen Verhältnisse in der Kupfererzlagerstätte von Mancayan auf Luzon (Philippinen): *Neues Jahrb. f. Mineral. Geol. u. Paleon.*, Beilage Band, vol. **56**, Abth. A, pp. 316–333, 1927.

[3] See p. 560.

The forms present on the small crystals of sulvanite are the cube *a*(100), the dodecahedron *d*(110), and the octahedron *o*(111). Of these, the cube is dominant on all crystals. The usual combination is *a*, *d*, small triangular faces of *o*(111) being observed on only two crystals. Some of the faces of *d*(110) are not developed and the faces on a single crystal vary in width, though all are much subordinate to the large faces of the cube *a*(100).

Nearly all the faces of the measured crystals gave poor reflections due to their dullness, sulvanite being appreciably attacked by dilute acids. On the matrix, the natural faces of the crystals are bright as are also the cleavage surfaces, which in part are minutely step-like.

The angles measured on three crystals from Utah are as follows:

MEASUREMENTS OF ANGLES ON CRYSTALS OF SULVANITE FROM UTAH

	$a \wedge a'$	$a \wedge d$
	89° 19′	44° 17′
	89 37	44 25
	89 51	44 26
	89 55	44 39
	89 56	44 40
	90 20	44 47
	90 23	45 01
	90 31	45 07
	90 59	45 59
Av.	90 06	44 49
Calc.	90 00	45 00

The small triangular faces of *o*(111) were so dull that they gave no reflections and were not measured.

It might be contended that the evidence presented does not preclude the possibility of the crystals being orthorhombic. The slight elongation of the crystals, the development on some of them of only one or two cleavage traces, and the absence of all the faces of the dodecahedron on a single crystal suggests a lower symmetry than that of the isometric system. The angular measurements could also be referred, for example, to those of an orthorhombic mineral like anhydrite:

Measured on sulvanite	Anhydrite
$a \wedge a' = 90^\circ\, 06'$	$a(100) \wedge c(001) = 90^\circ\, 00'$ $a(100) \wedge b(010) = 90\;\; 00$ $b(010) \wedge c(001) = 90\;\; 00$
$a \wedge d = 44\;\; 49$	$b(010) \wedge s(011) = 44\;\; 59$

However, the distribution of the faces of $d(110)$ on the crystals of sulvanite was not that of an orthorhombic crystal. The three rectangular cleavages and the angular measurements, moreover, do not agree with those of enargite, with which sulvanite should be analogous if it were orthorhombic.

Examination of a polished surface of sulvanite from Utah, by Dr. M. N. Short[4] has shown that the mineral is isotropic. Orcel[5] apparently was the first to note that in reflected polarized light sulvanite was strictly isotropic and he therefore considered it to be isometric. A few months later in the same year de Jong[6] published his conclusion, also finding it to be isotropic.

CHEMICAL COMPOSITION

An approximate analysis (by Schempp) gave the values shown in column 1. The two analyses by Goyder[7] (2 and 3) and the two analyses by Schultze[8] (4 and 5), all made on sulvanite from Australia, are here reproduced, with the unessential constituents deducted and then recalculated to 100 per cent. The analysis by Ir. J. deVries,[9] also on material from Australia, is given under (6). Under (7) is given the theoretical composition calculated for the established formula Cu_3VS_4.

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[4] Personal communication.

[5] Orcel, J., Notes sur les caractères microscopiques des minéraux opaques, principalement en lumière polarisée: *Bull. soc. franc. Mineral.*, vol. **51,** pp. 205–206, 1928.

[6] de Jong, W. F., Die Enargitgruppe. Struktur des Sulvanit, Cu_3VS_4: *Zeit. für Krist.*, vol. **68,** pp. 522–530, 1928.

[7] Goyder, G. A., On sulvanite, a new mineral: *Journ. Chem. Soc.*, vol. **77,** p. 1094, 1900.

[8] Schultze, H., and Steinmetz, H., Über Vanadium-Schwefelverbindungen; Inaug.-Dissert. (H. Schultze) Munich, 1908.
Abstr. in *Zeit. f. Krist.*, vol. **49,** p. 640, 1911.

[9] Quoted by de Jong, *Zeit. f. Krist.*, vol. **68,** p. 524, 1928.

ANALYSES OF SULVANITE

	Utah Schempp	Australia					Cu_3VS_4
		Goyder		Schultze		de Vries	
	1	2	3	4	5	6	7
Cu	50.0	51.57	52.96	51.89	52.66	51.20	51.55
V	14.5	13.46	13.72	13.08	13.40	14.20	13.78
S	30.0	34.97	33.32	35.03	33.94	34.60	34.67
	94.5	100.00	100.00	100.00	100.00	100.00	100.00

The determination of sulphur in column 1 is probably several per cent too low, as indicated by the low summation. The five analyses of sulvanite from Australia are in very close agreement with the calculated percentages for the formula Cu_3VS_4.

Sulvanite being isometric and not orthorhombic, as the chemically corresponding mineral enargite, Cu_3AsS_4, it may be questioned whether the formula of sulvanite should not be written $Cu_{12}V_4S_{13}$, following the formula of isometric tetrahedrite, $Cu_{12}Sb_4S_{13}=5Cu_2S\cdot 2CuS\cdot 2Sb_2S_3$, as written for example by Wherry and Foshag[10] but the analyses of sulvanite from Australia do not support this view.

Sulvanite, then, is chemically analogous to enargite and famatinite but crystallographically it is distinct, these three minerals being representatives of the type formula: Cu_3XS_4, the relationships being as follows:

	ISOMETRIC	ORTHORHOMBIC
Cu_3AsS_4	Green enargite[a]	Pink enargite
Cu_3SbS_4	......	Famatinite
Cu_3VS_4	Sulvanite	......

[a] See de Jong, *loc. cit.*, p. 522. Also Schneiderhöhn, H., *Anleitung z. mikro. Bestimmung von Erzen*, **1922**, p. 240.

OTHER LOCALITIES

The reported occurrence of sulvanite in Argentina has been the cause of some controversy. The occurrence was described by

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[10] Wherry, E. T., and Foshag, W. F., A new classification of the sulfo-salt minerals: *J. Wash. Acad. Science*, vol. **11**, p. 6, 1921.

Wiedemann[11] but the identification of sulvanite has been questioned by Frebold[12] who regards the mineral, referred to as sulvanite by Wiedemann, to be galena. Frebold's conclusion was later refuted by Wiedemann.[13]

In view of the determination of the isometric symmetry, with cubic cleavage, of the sulvanite from Utah, and the isotropic character of the mineral from Australia as determined by Orcel, de Jong, and Short, the objections raised by Frebold do not seem to hold. Wiedemann described his material as having a dark cream yellow color on polished surfaces whereas Frebold mentions a white color. The examination of sulvanite from Australia and from Utah, by Short,[14] shows the mineral to have a galena-white color on polished surfaces in reflected light.

Although the occurrence of sulvanite in Argentina should be confirmed, it does not seem justifiable, on the basis of Frebold's remark, to discredit the identification of Wiedemann.

The fourth recorded occurrence of sulvanite, from Siberia, is given by van der Veen,[15] without any description and is questioned by Schneiderhöhn and Ramdohr.[16]

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[11] Wiedemann, H. H., Geologische und bergmännische Untersuchung der Vanadinlagerstättin in der Sierra de Cordoba, Argentinien: *Mitteilungen des Roemer-Museums zu Hildesheim*, Nr. **28**, Mai, 1927. The paragraph relating to sulvanite (p. 12) is reproduced by Wiedemann in *Centralbl. Mineralogie*, **1928**, Abt. A, pp. 293–294.

[12] Frebold, Georg, Über das Vorkommen des Sulvanits (Cu_3VS_4) in der Sierra de Cordoba, Argentinien: *Centralbl. Mineralogie*, **1928**, Abt. A, pp. 27–28.

[13] Wiedemann, H. H., *Centralbl. Mineralogie*, **1928**, Abt. A, pp. 293–294.

[14] Personal communication. See also: Short, M. N., Microscopic determination of the ore minerals: *U. S. Geol. Survey*, *Bulletin* No. **825**, p. 92, 1931. Short describes the Australian sulvanite as isometric, color galena-white, cubic cleavage, not prominent.

[15] van der Veen, W. R., *Minerography and ore-deposition*. The Hague, **1925**, p. 111. (Fig. 126).

[16] Schneiderhöhn, H., and Ramdohr, H., *Lehrbuch d. Erzmikroskopie*, **1931**, p. 460.

NOTES ON SOME MINERALS FROM THE RHODOLITE QUARRY NEAR FRANKLIN, NORTH CAROLINA

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INTRODUCTION

Near the summit of Mason Mountain, on the southern slope, about 8 miles north of Franklin, North Carolina, is an old quarry from which rhodolite garnet has been collected. Abandoned for many years, this quarry was recently re-opened by Mr. Burnham S. Colburn of Biltmore, N.C., but after a number of attractive specimens of rhodolite had been obtained operations were discontinued. Excellent samples of rock which contain much gedrite are still to be found on the dump which, however, has been so carefully picked over for rhodolite that good samples are scarce.

The aggregate of minerals is very coarse grained and there is considerable variation in the proportion of the contained minerals so that hand samples differ greatly in appearance. A chemical analysis of such a mixture would be of little value unless the sample analyzed was representative of the quarry face. The essential minerals are rhodolite, gedrite, hypersthene and biotite. Small masses of quartz are present but this is not an important constituent. The following estimate of the quantity of each mineral present was made upon a polished sample approximately half of a square foot in size.

Rhodolite	55–60 per cent
Gedrite	15–20 " "
Hypersthene	15–20 " "
Biotite	10–15 " "

GEOLOGY

The rock containing these minerals occurs in a dike-like mass some 15–20 feet wide, striking in a northerly and southerly direction with an almost vertical dip. The schists immediately in contact on both the east and west are badly altered and decomposed. The dike, however, is perfectly fresh and has completely escaped the attack of weathering agents. It is difficult to closely compare these badly altered schists but the weathered exposures suggest that there is a slight difference in the direction of the schistosity. The schist to the west contains a greater quantity of large garnets than that to the east and according to verbal reports from some of the workmen employed during the recent operations, several rather good pink garnets were found in this rock. Such minor differences suggest that the rhodolite-gedrite rock has been intruded along a fault where there has probably been some displacement.

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The presence of hypersthene and garnet suggests that the original rock probably had a composition somewhat similar to eclogite, but the present aggregate containing gedrite, indicates a later metamorphism and perhaps deeper along the dike a normal eclogite may be found.

MINERALOGY

The minerals described in the following pages were extracted from material No. 92684, U. S. National Museum.

RHODOLITE. This variety of garnet was first brought to the attention of the public by A. M. Field of Asheville and Kunz[2] states: "Mr. Field also found 118 garnets worth $117.00 in Burk and Mason County, North Carolina. The value, per carat, was from $1 to $10.00." The first specimens were found in Cowee Creek and other streams originating in Mason Mountain, the gravels of which were worked by hydraulic processes. W. E. Hidden and J. H. Pratt[3] described this garnet and suggested the name *rhodolite* from two Greek words, one meaning rose and the other, stone. Such a name is particularly appropriate since the color of this garnet varies much the same as do the rhododendrons which are so common in the district. The color is pale rose pink on the exposed surface, but when freshly broken these garnets often show a distinct purplish tint. These same authors[4] described the minerals associated with the rhodolite in the stream beds and referred to a ledge of rock exposed near the summit of Mason Mountain which was composed almost entirely of rhodolite and biotite with a considerable quantity of disseminated iron sulphides. This description does not apply to the exposure visited by the writer with Mr. William Colburn, in that the sulphides in this quarry occur in very limited quantities.

The masses of rhodolite vary considerably in size; in Mr. Colburn's collection there are several specimens with a maximum

[2] Mineral Resources of the United States, 1893, *U. S. Geol. Survey*.

[3] *Amer. Jour. Sci.*, vol. 5, 1898, p. 294.

[4] *Amer. Jour. Sci.*, vol. 6, 1898, p. 463.

cross section of over two inches. Euhedral garnet crystals from this place are apparently quite rare, if not unknown, for in the visit to the quarry none was seen nor are they represented in the mineral collections examined. The average size of these garnet masses is probably between one and two centimeters. The rhodolite is badly fractured, but small pieces are perfectly transparent and free from mineral inclusions. In a number of thin sections the rhodolite is seen inclosing irregularly bounded small fragments of hypersthene in parallel orientation.

The following two analyses of this garnet have been published.[5]

Table I

Rhodolite from Mason's Branch, North Carolina

J. H. Pratt, Analyst

	I	II	III	Average	Ratios	
SiO_2	41.54	41.65		41.59	.689	.689=2.90
Al_2O_3		23.26	23.10	23.13	.226	.237=1
Fe_2O_3	1.88	1.92		1.90	.011	
FeO	15.48	15.63		15.55	.216	.659=2.78
MgO		16.90	17.48	17.23	.427	
CaO	1.02	.86		.92	.016	

Specific gravity 3.837–3.838.

The composition was found by calculation to correspond to two molecules of pyrope and one of almandite.

A ferrous iron determination was made upon the recently collected rhodolite for comparison with Pratt's results. An average of two determinations gave 16.06% FeO, which is only 0.51% higher than the value previously given. With such close agreement, it is evident that this garnet is the same as that described by Hidden and Pratt.

The index of refraction was determined to be 1.758 and the index given by Larsen for the originally described rhodolite is 1.76.

Gedrite. Gedrite is the most interesting mineral in this aggregate. It occurs in coarse prismatic crystals some of which measure 15 mm. long and 5 mm. wide. It is almost transparent and has a bright clove brown color.

The following analysis and optical study was made upon a pure sample prepared by handpicking. The indices of refraction are: $\alpha=1.642$, $\beta=1.655$, $\gamma=1.661$, all $\pm .003$, birefringence .019, optical character (−), axial angle, large. The molecular ratio for SiO_2 exactly equals the total ratios for the RO constituents, such close agreement being unusual in gedrites. Most of the analyses of this mineral give ratios which differ considerably from the normal 1:1 ratios for these constituents. The Al_2O_3 content is higher than that reported in any of the described gedrites and is molecularly almost one third that of either SiO_2 or RO.

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[5] *Op. cit.*

Table II

Gedrite from Mason's Mountain

E. P. Henderson, Analyst

Analysis		Ratios	
SiO_2	44.22	.7333	.7333
Al_2O_3	23.79	.2328	.2328
FeO	9.21	.1282	
CaO	0.62	.0110	
MgO	20.69	.5131	.7334
MnO	0.16	.0023	
H_2O	1.42	.0788	
Fe_2O_3	0.20		
	100.31		
Sp. Gr. 3.178			

The above analysis can be expressed in the following manner:

	Per cent
$FeSiO_3$	16.94
$MgSiO_3$	51.63
$CaSiO_3$	1.28
H_2SiO_3	6.18
$MnSiO_3$	0.29
Al_2O_3	23.79
Fe_2O_3	0.20
Total	100.31

When the molecular ratios of the gedrite from Mason Mountain are compared with the ratios of the rhodolite (see Table III), it was found that they are very similar.

TABLE III

	Rhodolite, Mason's Branch, North Carolina	Gedrite, Mason Mountain, North Carolina
	Ratios	Ratios
SiO_2	.689=2.90	.7333=3.14
R_2O_3	.237=1.	.232 =1.
RO	.659=2.78	.7334=3.14

From the above table it can be seen that the ratios in gedrite are essentially 3:1:3, yielding the type formula of the garnets. In fact the ratios come as near having the garnet formula as those of the rhodolite. However, in the gedrite there are certain minor elements grouped under RO, such as water and alkalies, which are not found in the garnets. The rhodolite molecularly contains approximately one third as much of the iron garnet as it does of the magnesium garnet. The gedrite also contains about one third as much of $FeSiO_3$ as it does of $MgSiO_3$. It is unusual to find two of the essential constituents of a rock having such close similarity in their molecular proportions.

Evans[6] described a gedrite from Haliburton County, Ontario, which occurs with a garnet but he makes no reference to the composition of the garnet.

HYPERSTHENE. This mineral has a greenish-gray color and occurs in small masses the larger ones scarcely exceeding two centimeters in maximum direction. Frequently there are small inclusions of biotite in the hypersthene.

The following analysis and indices of refraction were determined upon a carefully selected sample.

$$\alpha=1.685,\ \beta=1.696,\ \gamma=1.699, \text{ all } \pm .003;$$

birefringence .014, optical character (−). These ratios conform to the general hypersthene formula although the discrepancy between the RO and SiO_2 ratios is slightly greater than it should be. No attempt was made to place the Al_2O_3 in the formula. However, if it is added to the SiO_2 the ratio between the $SiO_2+Al_2O_3$:RO becomes .9103:9040, and this is nearer to the normal 1:1 ratio for these elements. The molecular proportion between FeO and MgO is 1:2.73.

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[6] *Amer. Jour. Sci.*, vol. **25**, 1908, p. 511. This analysis was found, by calculation, to be considerably different from the normal ratios of a gedrite and no indices of refraction were given so it could not be compared with the North Carolina material.

Table IV

Hypersthene from Mason's Mountain

E. P. Henderson, Analyst

Analysis		Ratios	
SiO_2	52.36	.8683	= .8683
Al_2O_3	4.29	.0420	= .0420
FeO	16.90	.2352	= .8851
CaO	0.44	.0078	
MgO	25.89	.6421	
H_2O	0.34	.0189	
	100.22		

Biotite. The black mica is considered to be a biotite; it may, however, be a phlogopite. Nothing particularly unusual was noted about it and since the purpose of this paper is to discuss the relationship of gedrite and rhodolite, a complete chemical study of the mica was not undertaken.

This mica occurs in coarse irregular plates some of which measure two and one half centimeters on a cleavage face. The color is very dark brown to black when seen in the matrix but thin cleavage sheets are amber-brown. The FeO content is 5.78% and the Fe_2O_3 content is 0.72%. The indices of refraction are: $\alpha=1.565$, $\beta+\gamma=1.607$, all ±.003, optical character (−), 2E is 15°.

URANINITE FROM THE HURON CLAIM, WINNIPEG RIVER AREA, S. E. MANITOBA*

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It has been demonstrated in recent years that eastern Manitoba has a wealth of pegmatites containing a wide variety of rare minerals and elements. The rock formations in which they occur are widely separated from the Precambrian bodies farther east, which have been named and classified.

The pegmatites occur in both plutonics and associated roof-pendants of metamorphosed volcanics and sediments. The latter occurrences are more likely to contain rare elements. Dykes and other forms or pegmatite, seemingly of the same general character and with some or all of an assemblage of rare minerals, are found as far south as Shoal lake, near the Ontario boundary of southeast Manitoba, and as far north as Grass river, beyond the Hudson Bay railway. They are most abundant, so far as is known, between and near Oiseau and Winnipeg rivers and to the south near West Hawk and Falcon lakes. Some have been found near Island lake.

The association of rare minerals, in whole or in part, which appears to permit a correlation over wide areas, consists of the lithium minerals (spodumene, lepidolite, and amblygonite), beryl, topaz, cassiterite, molybdenite, bismuthinite, bismuth, apatite, monazite and several lithium-bearing phosphates, columbite-tantalite and last among others, uraninite.

Uraninite was discovered during the summer of 1930 on the Huron claim. This is one of a large group of claims staked by the Winnipeg River Tin Company near Winnipeg river. The Huron claim is about a half mile inland from a point on the southeast shore of the river, nine or ten miles above Pointe du Bois. It is believed to be the first discovery of the mineral in Manitoba. The pegmatite body carrying the uraninite lies in and is parallel to the strike of a roof-pendant of andesite schist. The schist has an east-west strike parallel to a granite contact about 1,500 feet to the north.

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There are seven or eight outcrops of pegmatite in a surface depression which is largely filled by a thick accumulation of drift. Contacts of pegmatite and schist have been exposed on either side of the depression. They dip away from the depression at low angles on each side, thus suggesting that the several outcrops are joined into a single dyke in the form of an anticlinal arch or dome. The top of the arch has been eroded.

In two or three of the outcrops of pegmatite on the Huron claim, notable segregations of beryl were encountered. Further work led to the finding of tantalite-columbite and finally of monazite and uraninite in one of the outcrops.

The most prominent mineral in the pegmatite is a reddish-weathering feldspar. Quartz is also prominent and shows an unusual degree of segregation from the feldspar, appearing in more or less pure masses. Beryl is prominent and is associated with both quartz and feldspar. In the one outcrop, which so far has yielded uraninite, this mineral together with monazite and tantalite occur as crystals and grains embedded in feldspar.

Monazite is more abundant than uraninite and if it has a sufficiently high content of thorium, a determination of the lead ratio will be of great interest for comparison with that from the uraninite.

Composition

The material as received from DeLury consisted of several pounds of pegmatite fragments composed chiefly of pinkish albite with some yellowish beryl and grayish-green radiating fibrous zoisite. The uraninite occurs sparingly as small crystals of cubic habit from one eighth to one quarter inch diameter embedded in the feldspar. On examination under the binocular some of the uraninite crystals appeared to be in a very good state of preservation, being hard, grayish steely black and with uneven fracture. Others, however, were more altered, some to the pitch-black stage of uraninite alteration, some showing outer rims and cracks with yellowish alteration products.

The small amount of uraninite available did not permit a rigid hand selection of only the hardest material. The better crystals were broken out from the feldspar and the quantity of about 6 grams of crude material thus obtained was crushed on a steel plate, picked over to eliminate decomposition products as much as possible, then gently ground under water in an agate mortar, the slimes being discarded, until all passed 100 mesh. The 100 mesh product, about 4.5 g., was panned in a clean aluminum pan until a heavy fraction of slightly over 2 g. with Sp. Gr. = 8.082 at 21.70°C. was obtained. Two grams of this was used for analysis I. The rest, with the material remaining from the panning, having Sp. Gr. = 7.856, was subjected to further severe panning using as wash liquid dilute hydrochloric acid of approximately 17 per cent HCl content, at room temperature. This acid panning, which required several hours of manipulation, was carried out on large watch glasses and was very effective. The acid solution seems to have some effect on the surface tension which facilitates panning. All the feldspar grains were removed and much of the lighter black uraninite. If a larger quantity of mineral had been available it probably would have been possible to isolate an even heavier fraction of steely colored material by eliminating the black grains. The acid dissolved some uranium during the first few minutes of treatment with a slight momentary evolution of gas, probably either CO_2, or helium or both, from minute films of soluble secondary minerals. During the subsequent treatment of perhaps 3 hours or more duration the acid remained practically colorless. The final heavy product stood in acid of this strength for an hour or two without producing visible coloration. After very thorough, prolonged washing, the concentrate was found to have Sp. Gr. = 8.968 and the total amount of 1.4 g. was used for Analysis II.

CHARACTER OF THE ANALYZED MATERIAL

Lot No. I obtained by panning the whole original quantity in water, appeared under the microscope to be first class steely to black material free of yellow or orange decomposition products, but containing a few grains of feldspar.

Lot No. II similarly consisted of steely to black grains entirely free of feldspar and colored decomposition products.

It might be added in view of the high lead results obtained, that there is not the least suspicion that galena or any other foreign minerals were present. The material used for analysis was to all appearances just as good as that of numerous other uraninites previously analyzed by the writer.

As only 2 g. was available for analysis I and 1.4 g. for II, it was considered advisable to use the whole amount in each case for the main determinations, so that UO_2, H_2O, etc. were not determined.

The analytical results are as follows:

Uraninite, Huron Claim, Winnipeg River District, S. E. Manitoba
H. V. Ellsworth, Analyst

The Analysis of Uraninite from S. Dakota by C. W. Davis is given in part for comparison under III.*

	I	II	III
	Manitoba Uraninite, Heavy fract. of Water Concentrate	Manitoba Uraninite, Acid treated Concentrate	Uraninite from Ingersoll Claim, Black Hills, S. Dakota
PbO	16.63	16.71	16.42
(Pb)	(15.44)	(15.50)	(15.24)
UO_2	—	—	48.87
UO_3	—	—	28.58
U_3O_8	63.08	64.86	—
(U)	(53.50)	(55.01)	(66.90)
ThO_2	14.18	13.94	2.15
(Th)	(12.46)	(12.25)	(1.89)
(Th×0.38)	(4.73)	(4.65)	(0.72)
$(Ce, La, Di)_2O_3$	0.37	0.28	1.06
$(Yt, Er)_2O_3$	1.02	1.19	1.01
Fe_2O_3	0.64	0.75	0.30
MnO	0.18	0.13	0.001
Al_2O_3, etc.	0.10	0.12	—
CaO	1.45	1.72	0.46
MgO	0.07	0.06	0.01
SiO_2	0.67	0.37	0.05
Insol. feldspar, etc.	0.12	none	0.15
H_2O, He, etc.	not det.	not det.	H_2O 0.44
Sp. Gr.	8.082	8.968	9.182
$\frac{Pb}{U+0.38\ Th}$	0.265	0.260	0.225

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* *Am. Jour. Sc.*, March, 1926.

ANALYTICAL NOTES

The lead, uranium and thorium precipitates were very carefully checked for impurities. The lead is, if anything, slightly low due to exceptional precautions having been taken to ensure a pure product.

The manganese content is unusually high for uraninite, in the writer's experience.

Summations of uraninite analyses do not mean much unless all constituents such as UO_2, UO_3, H_2O, CO_2, He, etc. have been determined. The low total of Analysis I, however, probably indicates that an unusually large amount of H_2O and probably CO_2 were present in minute films of alteration products which were removed by the acid treatment from the material of Analysis II.

Geological Age Relationships

The Huron claim uraninite is remarkable for its high content of lead and thorium, and its very high lead ratio which makes it the oldest uraninite known so far as the writer is aware. The question immediately arises as to whether the age indications can be accepted as reliable. So far as the quality of the material analyzed is concerned, it appeared to be fully as good as that of some other Canadian Precambrian uraninites previously analyzed which have yielded lead ratios of 0.15 to 0.16. Further, the high percentage of ThO_2 in the Manitoba uraninite is not altogether exceptional as the Wilberforce, Ontario, uraninite has nearly the same thorium content and yet yields a lead ratio between 0.15 and 0.16. The high lead ratio of the Huron claim uraninite is closely approached by that of only one other known uraninite, that from the Ingersoll claim, South Dakota, analyzed by C. W. Davis, the essential figures for which are reproduced in the third column of analyses above.[1] It is significant that both uraninites occur in the same general region in pegmatites of a very similar, one might say identical, type characterized by the presence of large amounts of albite and lithium and beryllium minerals. It has been claimed by Hess and Schaller that the albite in such pegmatites is a replacement on a large scale of earlier formed minerals during the later phase of magmatic activity. If earlier formed minerals including possibly uraninite have been reworked in this way, one might wonder what is the effect of such alteration and replacement on the uraninite. Schaller and Hess, however, believe that uraninite and other rare element minerals occurring in albite pegmatites do not belong to the first generation of minerals but were formed during the albitization process. That uraninites occurring in albite pegmatites do not on that account yield abnormal lead ratios is supported by the fact that uraninites of the albitic lithium-bearing New England pegmatites yield comparatively small lead ratios in complete accord with the age as determined by ordinary geological methods. Thus, there seems to be no reason to doubt the age indications of the Dakota and Manitoba uraninities on the ground that a special sort of alteration or replacement associated with albitization and lithium mineralization has increased the lead content. The only other doubts which may enter result from the lack of exact knowledge as to the origin of actinium and the possibility of the existence in the early stages of the earth's history of short-lived isotopes of uranium. The older idea of physicists that ordinary lead occurs as a constituent of uraninites has been pretty well exploded by recent work, and has always seemed most improbable to mineralogists.

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[1] It is true that some of the specimens of uraninite from Sinyaya Pala, Carelia, Russia, have shown even higher lead ratios, but these are described as being contaminated either by galena or by red and yellow alteration products such as gummite. The purest and freshest specimen analyzed yielded the lead ratio 0.17.

The rocks of the area in which the Huron claim pegmatite occurs have been studied and mapped in detail by J. F. Wright[2] who states that the oldest rocks there are a complex of ancient Precambrian lavas and sediments named by him the Rice Lake series. The lavas comprise basalt, andesite, dacite and rhyolite. The sediments include quartz-mica-garnet gneiss, mica schist, quartzite, arkose and slate, with some conglomerate beds carrying granite pebbles. In places the lavas and sediments are interbedded but large areas are occupied mainly by either volcanics or sediments. No evidence of an unconformity between the different members of the complex has yet been found.

The Rice Lake series is cut by intrusives ranging in composition from peridotite to granite, all believed to be closely related in origin and of about the same age. The most abundant of these intrusives are granites and diorites and especially microcline granite. Four types of granitic bodies have been distinguished by Wright: (1) Diorite and granodiorite; (2) Granodiorite and quartz diorite; (3) Pink microcline granite; (4) White and pink pegmatitic albite granite. All these are believed to be differentiates from the same magma. The earlier more basic types are sometimes cut by the later more acid differentiates. The albitic lithium-bearing pegmatites are associated with the albite granite.

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[2] *Geol. Sur. Canada,* Summary Report, 1924, Part B, p. 51.

The Rice Lake series has not yet been definitely correlated with the classical Keewatin-Couchiching rocks to the eastward in Ontario but both are evidently very similar lithologically.

If the age indication of the Huron claim uraninite is accepted, the Rice Lake series must comprise some of the oldest rocks of the earth's crust and the granitic rocks and pegmatites which cut them are much older than the granites and pegmatites which cut the Grenville series in Old Ontario and Quebec.

Further, taking as limits the Huron claim uraninite with ratio 0.26 and the Henvey uraninite, which is early Precambrian with ratio 0.11, the interval of Precambrian time thus defined appears to comprise at least three fifths of all geological time.

NOTE—In comparing the Manitoba and Dakota uraninites it may be noticed that the lead percentages are much the same in both, also the total U and Th of the Manitoba uraninite is about the same as the U of the Dakota. It may be only a remarkable coincidence, but possibly there is some significance attached to the fact that if the Th of the Huron claim uraninite be calculated as U replacing it in molecular proportions, i.e. $12.25\ \mathrm{Th} = 12.56\ \mathrm{U}$, the resulting lead ratio of the Manitoba uraninite becomes 0.227, very close to that (0.221) of the South Dakota uraninite calculated in a similar way.

URANINITE FROM HENVEY TOWNSHIP, PARRY SOUND DISTRICT, ONTARIO

H. V. ELLSWORTH,* *Dept. of Mines, Ottawa.*

The occurrence of uraninite with thucholite, cyrtolite, oily hydrocarbons, etc., at the Besner mine, lot 5, conc. B, Henvey township, Parry Sound district, has been described in detail by H. S. Spence.[1] The writer's thanks are tendered Mr. Spence for contributing much of the material used in this study.

The Besner dike is not fully exposed, being almost wholly covered by overburden except for the actual opening which is about 100 ft. long, by 35 ft. wide, by 20 ft. deep. It strikes slightly north of east and cuts a coarse porphyritic granite gneiss named by T. T. Quirke the Dunlop porphyry, which covers a considerable area in this locality. The middle portion of the dike consists chiefly of pink, more or less perthitic microcline with masses of white quartz. Several carloads of commercial feldspar were shipped. Plagioclase feldspar, possibly albite, occurs along the south wall to a width of 2 or 3 feet in places. Large, thin, books or crystals up to 18 inches in diameter of more or less altered black mica are rather common.

The uraninite is very intimately associated with thucholite which appears to have partly replaced the uraninite in some instances. Even the best of the uraninite is more or less penetrated by microscopic veins or cracks filled with thucholite. In order to obtain material suitable for analysis about 50 grams of the best looking uraninite was selected for minimum thucholite content from five or six favorable crystals or masses. This was crushed on a steel plate to pass 20 mesh and any 80 mesh siftings were rejected. The 20 mesh lot (38 g.) was very lightly ground in an agate mortar and frequently sifted through 40 mesh until all was through. The material through 40 and caught on 80 was panned down to 18 g. and air dried. The specific gravity of this lot was 7.939 at 20.97°C. The minus 80 product was panned down to 4.6 g. and after air drying had a Sp. Gr. = 8.129 at 20.77°C. The 18 g. of −40 +80 product was further panned to 6.8 g. and after air drying had a Sp. Gr. = 8.173 at 20.67°C. This 6.8 g. lot was used for the analysis under I. The remainder of the 18 g. lot (about 11 g.) with Sp. Gr. = 7.827 at 23.55°C. was analyzed for lead, uranium and thorium with results given under II. The material of analysis III was about 5 g. from a single crystal completely enclosed in feldspar, but containing traces of thucholite. This was crushed and panned down to a heavy fraction of about 2 grams with Sp. Gr. = 7.809 at 18.5°C. which was used for the analysis. Clean aluminum pans and watch glasses were used to avoid contamination.

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* Published by Permission of the Director of the Geological Survey, Canada.

[1] This Journal, November, 1930.

The object of the foregoing operations was to get rid of as much as possible of the small amount of thucholite present and to obtain a concentrate of the freshest material by taking advantage of the fact that uraninite is harder, heavier and less friable, the less altered it is.

The analyses, carried out on 2 g. portions, yielded the following:

HENVEY TOWNSHIP URANINITE
H. V. Ellsworth, Analyst

	I	II	III
	Concentrate Sp. Gr. = 8.173	Concentrate Sp. Gr. = 7.827	Heavy fract. of single crystal Sp. Gr. = 7.809
PbO	8.66	8.09	8.16
(Pb)	(8.04)	(7.51)	(7.57)
UO_2	49.35*		
UO_3	29.76*		
(U_3O_8)	80.51	79.30	79.35
(U)	(68.29)	(67.26)	(67.30)
ThO_2	1.78	1.73	2.11
(Th)	(1.56)	(1.52)	(1.85)
$(Th \times 0.38)$	(0.59)	(0.58)	(0.70)
$(Ce, La, Di)_2O_3$	1.47	2.87	3.48
$(Yt, Er)_2O_3$	1.48		
Fe_2O_3	0.88		
MnO	0.02		0.02
Al_2O_3, etc.	0.17		
CaO	2.69	2.82	1.97
MgO	0.03		
CuO†	0.03	0.03	0.03
SiO_2	1.36	1.76	2.12
H_2O	1.44		
	99.12		
$\frac{Pb}{U+0.38\ Th}$	0.117	0.111	0.111

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* Owing to the presence of traces of thucholite carbon, UO_2 could not be determined by the usual method. The results cited were obtained by allowing the powdered uraninite to stand for 175 hours in HF at room temperature. Uranous fluoride

CHARACTER OF THE URANINITE

The physical properties of the Henvey uraninite, such as its low specific gravity, its pitch-black color, conchoidal fracture, relative softness (H = 5.5) and friability, all indicate that the mineral is in the second stage of alteration as described in an article[2] on the Villeneuve uraninite in which the mineral has altered from its original steel gray color with irregular fracture to a pitch black form with conchoidal fracture and lower density.

The comparatively easy solubility of the Henvey uraninite in hydrochloric acid confirms this physical evidence of alteration from the chemical side. It is probable that the analytical determination of UO_2 is in error and that the percentage found is too high.

AGE RELATIONSHIPS

Fortunately, if the evidence of the work cited on the alteration of the Villeneuve uraninite may be accepted as applying to uraninites in general, the alteration of the Henvey uraninite apparently has not gone far enough to render the lead ratio unreliable and it would presumably need reduction only in the third place of decimals. Considerable evidence has accumulated that altered uraninites in general do give slightly higher results than the freshest material from the same sources. On the other hand it is always possible that an exceptional type of alteration may occur in some instances. Nothing is known exactly as to the effect of the sort of alteration accompanying replacement by thucholite. It may be noted, however, that thucholite itself from the original Parry Sound occurrence[3] yields an extremely small and entirely erroneous lead ratio, from which it may be inferred that thucholitic alteration would possibly tend to diminish the lead ratio of uraninite. The re-

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is insoluble and is almost unaffected by air at ordinary temperature when present in the bottom of a dish with supernatant HF solution while uranic fluoride is soluble and can thus be separated. Decomposition appeared to be complete but the figure for UO_2 seems high considering the physical properties of the mineral. The presence of undecomposed mineral particles would increase the apparent percentage of UO_2.

† Copper was found in all the samples in about the same amount, and is certainly not an accidental impurity. This is the first uraninite in which the writer has detected this element.

[2] Four Stages in the Alteration of the Villeneuve Uraninite. This Journal, Oct. 1930.

[3] This Journal, Aug. 1928.

sults for the three Henvey samples analyzed seem at first sight to give some slight confirmation of this idea, as the heaviest fraction No. I yields a slightly higher lead ratio than the others. No. I contained scarcely more than traces of thucholite, but Nos. II and III contained small though appreciable amounts of thucholite, which because of its own low lead ratio would tend to lower slightly the lead ratios of the uraninite with which it is associated. However, the differences in the lead ratio of the three samples are small after all and the concordance of results from the two fractions of a 50 gram average lot and a single crystal is reassuring. It seems reasonable to assume that the true age of the uraninite is indicated by some value between 0.11 and 0.12 for the lead ratio, and for the present the highest value 0.117, which was derived from the best material, may be assumed to be most nearly correct.

Dr. W. H. Collins has very kindly contributed the following summary of the latest geological data relating to the area in which the Henvey pegmatite occurs:

"The Henvey uraninite-bearing pegmatite lies in the Key Harbour quadrangle, recently mapped for the Geological Survey by T. T. Quirke (see *Key Harbour Sheet*, No. **239A**). This area is underlain by paragneisses intruded by batholithic bodies of granite from which the pegmatite is believed to be a magmatic differentiate and therefore of practically the same age. The granites are placed in the Killarnean (the latest known Precambrian batholithic intrusion) by Quirke on the ground that the paragneisses which they intrude are metamorphosed Huronian sediments. On the other hand, all the granites of proven Killarnean age are characterized by a higher percentage of potash than of soda, whereas the granite at Henvey contains more soda than potash. In this respect the Henvey granite is more like the batholithic rocks of the Grenville sub-province, from which the uraninites have uniformly yielded lead ratios of 0.15 to 0.16. It is therefore not certain that the Henvey granite is Killarnean. Efforts have failed so far to obtain uraninite from pegmatite of undoubted Killarnean age, from which a reliable lead-uranium ratio could be obtained."

THE CRYSTAL STRUCTURE OF POTASSIUM DITHIONATE, $K_2S_2O_6$[1]

M. L. HUGGINS AND GLENN O. FRANK,[2] *Stanford University.*

Potassium dithionate crystallizes[3] with the symmetry of the trigonal trapezohedral class, D^3 $(A_3 \cdot 3A_2)$, of the hexagonal system. The observed forms include $\{0001\}$, $\{10\bar{1}1\}$, $\{10\bar{1}0\}$, $\{11\bar{2}0\}$, and $\{11\bar{2}1\}$. The crystals rotate polarized light, as would be expected of crystals having this symmetry.

The crystals used in the present investigation were prepared by treatment of an aqueous solution of barium dithionate with potassium sulphate, filtering, evaporating, and recrystallizing from water solution.[4]

THE STRUCTURE ANALYSIS

The x-ray analysis was made by the oscillating crystal method, a horizontal beam of x-rays, consisting chiefly of the K radiation of molybdenum, striking a crystal which was oscillated around a vertical axis at constant angular velocity through an angle of 20 degrees. A photographic film was placed normal to the x-ray beam,

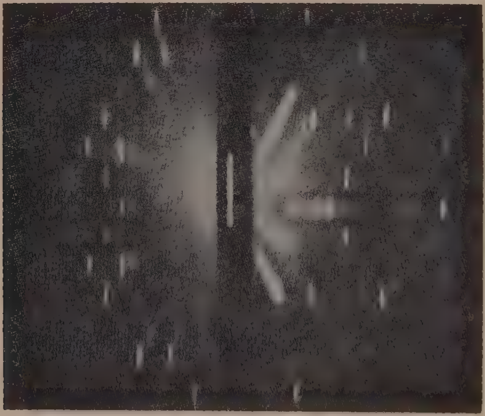

FIG. 1. Photograph taken with the crystal oscillating around the a axis.

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[1] This work was reported at the Toronto Meeting of the Mineralogical Society of America, December 30, 1930. A preliminary report, *Phys. Rev.*, **31**, 916 (1928), was incorrect in assigning the structure to the D_3^1 space group, the result of an error in calculation, but otherwise essentially correct.

[2] Now Mrs. Wallace Potter.

[3] Groth, *Chemische Krystallographie*, **2**, 690, 1908.

[4] Heeren, *Pogg. Ann.*, **7**, 55, 1826.

10 cm. from the crystal. One of the photographs obtained in this way is reproduced in Fig. 1.

For such a photograph the "identity distance," J, between two identical points within the crystal in the direction of the axis of rotation is obtainable from the wave-length λ and the angle μ_n between the central "layer line" (that of zero order) and that of order n by means of the relation[5]

$$J = \frac{n\lambda}{\sin \mu_n} .$$

In taking this photograph the axis of rotation was the a axis (as assumed by Groth[3]). Other photographs were taken with the crystal rotated about the c axis and about the axis a', perpendicular to c and making an angle of 30° with a. From these the following identity distances were obtained.

$$\begin{aligned} a &\backsim 9.8\text{Å} \\ a' &\backsim 17.0\text{Å} = \sqrt{3} \times 9.8\text{Å} \\ c &\backsim 6.4\text{Å} = 0.65 \times 9.8\text{Å} \end{aligned}$$

Since the identity distance in the direction of the a axis is less than that in the direction of the a' axis, the orientation as given by Groth gives the smallest unit cell. 9.8Å and 6.41Å are then approximately the unit distances a and c, i.e. the lengths of the edges of the unit cell.

More accurate values, obtained from the $(h0.0)$ and $(00.l)$ spectra, by comparison with reflections from a cleavage face of calcite, are

$$\begin{aligned} a_0 &= 9.82\text{Å} \\ c_0 &= 6.36\text{Å} \\ a_0 : c_0 &= 1:0.648. \end{aligned}$$

The axial ratio so obtained is in good agreement with that (1:0.6467) given by Groth.[3]

The volume of the unit cell, computed from these dimensions, equals 3.08 times the volume per "molecule," calculated from the density (2.278 g/cc.),[6] the molecular weight, and Avogadro's number. There are thus six potassium, six sulfur and eighteen oxygen atoms within the unit.

The determination of the indices of the observed reflections was simplified by making projections from the photographs with a ruler

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[5] Polanyi, *Naturwiss.*, 9, 337, 1921.

[6] *International Critical Tables.*

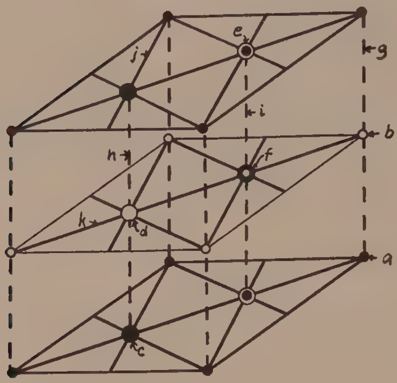

FIG. 2. The distribution of symmetry elements within the unit cell, for a structure having the symmetry of the space-group D_3^1 (from Wyckoff[9]).

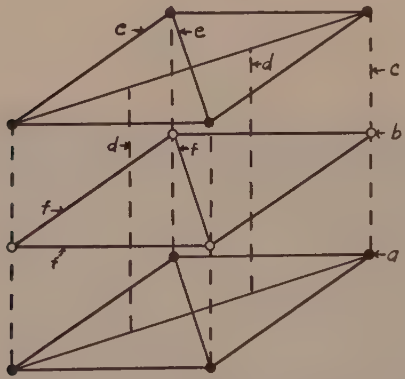

FIG. 3. The distribution of symmetry elements within the unit cell, for a structure having the symmetry of the space-group D_3^2 (from Wyckoff[9]).

devised by the senior author. The necessary data for constructing such a ruler and a description of its use will be published elsewhere.

The presence of many reflections having indices such that $(2h+k+l)/3$ is not integral proves the fundamental lattice to be the hexagonal rather than the rhombohedral.[7] This and the observation that $(00.l)$ reflections with $l/3$ not integral were obtained eliminate[8] as possibilities space-groups D_3^{3-7} inclusive, leaving only D_3^1 and D_3^2.

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[7] Schiebold, *Leipziger Abh.*, **36,** 65, 1919.

[8] Astbury and Yardley, *Phil. Trans. Roy. Soc.* (London), **A224,** 221, 1924. Wyckoff, *Zeit. Krist.*, **63,** 507, 1926.

The special distribution of symmetry elements within a unit cell of a structure isomorphous with the space-group D_3^1 is represented[9] in Fig. 2, the coördinates and symmetry of the various sets of equivalent positions being given in Table 1. Fig. 3 and Table 2 give the same for D_3^2.

TABLE 1

Equivalent positions for the space-group D_3^1 with the point-group symmetry for each.

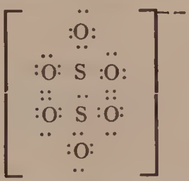
(a) D_3 000 (b) D_3 $00\frac{1}{2}$ (c) D_3 $\frac{1}{3}\,\frac{2}{3}\,0$ (d) D_3 $\frac{1}{3}\,\frac{2}{3}\,\frac{1}{2}$ (e) D_3 $\frac{2}{3}\,\frac{1}{3}\,0$ (f) D_3 $\frac{2}{3}\,\frac{1}{3}\,\frac{1}{2}$
(g) C_3 $00u;\ 00\bar{u}$ (h) C_3 $\frac{1}{3}\,\frac{2}{3}\,u;\ \frac{1}{3}\,\frac{2}{3}\,\bar{u}$ (i) C_3 $\frac{2}{3}\,\frac{1}{3}\,u;\ \frac{2}{3}\,\frac{1}{3}\,\bar{u}$
(j) C_2 $u\,\bar{u}\,0;\ 2\bar{u},\bar{u},0;\ u,2u,0$ (k) C_2 $u\,\bar{u}\,\frac{1}{2};\ 2\bar{u},\bar{u},\frac{1}{2};\ u,2u,\frac{1}{2}$
(l) C_1 $xyz;\ y-x,\bar{x},z;\ \bar{y},x-y,z;\ x,x-y,\bar{z};\ \bar{y}\,\bar{x}\,\bar{z};\ y-x,y,\bar{z}$

TABLE 2

Equivalent positions for the space-group D_3^2 with the point-group symmetry for each.

(a) D_3 000 (b) D_3 $00\frac{1}{2}$ (c) C_3 $00u;\ 00\bar{u}$
(d) C_3 $\frac{1}{3}\,\frac{2}{3}\,u;\ \frac{2}{3}\,\frac{1}{3}\,\bar{u}$ (e) C_2 $uu0;\ 0\bar{u}0;\ \bar{u}00$ (f) C_2 $uu\frac{1}{2};\ 0\,\bar{u}\,\frac{1}{2};\ \bar{u}\,0\,\frac{1}{2}$
(g) C_1 $xyz;\ y-x,\bar{x},z;\ \bar{y},x-y,z;\ y\,x\,\bar{z};\ \bar{x},y-x,\bar{z};\ x-y,\bar{y},\bar{z}$

Chemical evidence indicates that the substance contains $S_2O_6^{--}$ groups in which the sulfur atoms are bonded together and each sulfur is bonded also to three of the oxygens. The valence bonds (electron-pairs), according to the Lewis theory,[10] should be tetrahedrally disposed around the sulfur atoms, this arrangement being represented in two dimensions by the structural formula:



Much evidence from various experimental fields[11] might be cited to show that such an arrangement is very probably correct. In this work we have tentatively assumed it to be so, to see whether or not such an assumption will lead to a distribution of atomic centers in agreement with our experimental data.

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[9] Wyckoff, The Analytical Representation of the Results of the Theory of Space-Groups, *Carnegie Inst. Pub., No.* **318,** Washington, 2nd Edition, 1930, pp. 153, 154, 223, 224.

[10] Lewis, *Valence and the Structure of Atoms and Molecules,* Chem. Cat. Co., New York, 1923.

[11] Cf. Huggins, *Phys. Rev.,* **27,** 286, 1926.

The S–S centerline in such a group is a three-fold symmetry axis. Since the crystal also possesses three-fold symmetry axes, we should expect the sulfur atom centers to lie on them, that is, if D_3^1 is the correct space-group, in positions (g), (h) and (i). The oxygen centers would then be in three sets of positions (l), with such parameters as would place them similarly around each S_2 group. The six potassium atoms could either be in (l) also or in various combinations of the other positions. From other crystal structure data rough values of the S–S and S–O distances (2.1 and 1.5–1.6Å respectively) can be obtained. From these and the assumption that the angles between the bonds joining each sulfur atom with the four surrounding atoms are not far from 109.5° (the angle between the lines joining the corners of a regular tetrahedron with its center) were calculated approximate values for the three u parameters for sulfur and the three z parameters for oxygen. (Both structures in which the centers of all the negative ions are in the same plane, say $z=0$, and those in which two such ion centers are in one plane, say $z=0$, while the other is in a different plane, $z=\frac{1}{2}$, were of course considered). Then for the various alternative distributions of the K atoms structure factors were calculated for the first six orders of reflection from (00.1) planes, using the relationship[12]

$$F_{\text{calc}} = \sqrt{A^2 + B^2} \quad \text{where}$$

$$A = \sum_i F_i \cos 2\pi\,(hx_i + ky_i + lz_i)$$

$$B = \sum_i i\, F_i \sin 2\pi\,(hx_i + ky_i + lz_i).$$

The summations are taken over all the atoms i in the unit cell. F_i is the atomic structure factor for the ith atom (K, S or O). Various curves for the variation of these factors with sin θ were tried. In no case was even fair agreement obtained with the F_{obs} values (Table 3). The latter were obtained from the observed intensities, estimated by visual comparison with a scale produced by placing a film in the path of the direct beam for various known lengths of time, by means of the relation

$$F_{\text{obs}} = \sqrt{\left(\frac{\sin 2\theta}{1 + \cos^2 2\theta}\right) I_{\text{obs.}}}$$

(These F_{obs} values are of course only relative. Moreover no account is taken in their calculation of any correction for absorption, approximate calculations having shown that the error thereby introduced is insufficient to invalidate any of the conclusions reached. The chief effect of this neglect is to make F_{obs} somewhat smaller than it should be for very strong reflections).

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[12] Wyckoff, *The Structure of Crystals*, Chemical Catalog Co., New York, **1931**, p. 95.

The disagreement can be most simply illustrated by considering only the (00.*l*) reflections for which *l* is even. Using the structure factor curves given in Fig. 4, relative F_{calc} values for (00.2), (00.4) and (00.6) are (neglecting the sign) 10, 12 and 4 respectively, for all arrangements except those with the K atoms in positions (*l*). In those cases the magnitude of F_{calc} is (on the same scale) for all values of Z_k equal to or larger than 10 for (00.2), equal to or smaller than 12 for (00.4), and equal to or less than 5 for (00.6). These are to be compared with F_{obs} values of 4, 10 and 7, respectively. It is

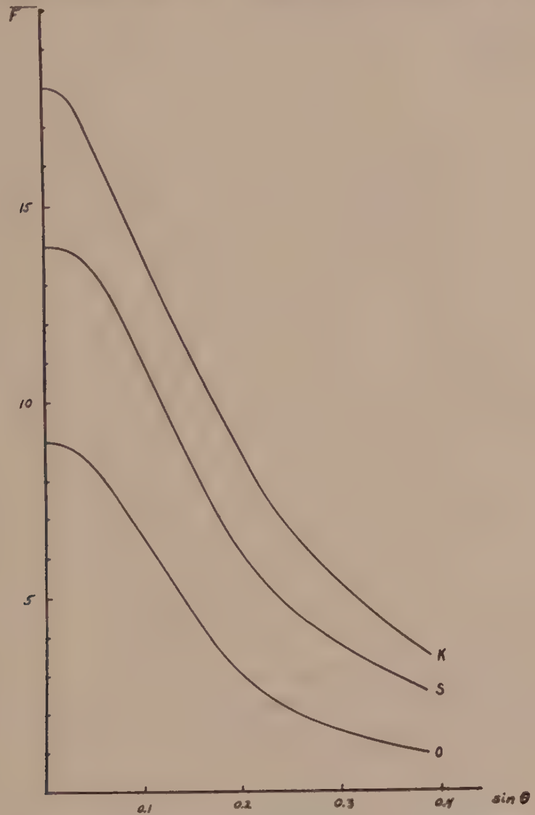

FIG. 4. Atomic *F* curves used in this structure analysis.

therefore evident that D_3^1 cannot be the correct space-group, provided our assumptions are even approximately correct.

A similar procedure was followed for the space-group D_3^2. (See Fig. 4 and Table 2). Good agreement with the observed intensities of the (00.*l*) spectra was obtained only for the case in which the *S* atoms are in pairs on the three-fold symmetry axes, i.e. in positions (c), (d) and (d), with the centers of these pairs in layers $z=0$, $z=v$ and $z=\bar{v}$, v being in the neighborhood of 0.42–0.43, and with the potassium atoms in positions (e) and (f), on two-fold symmetry axes.

Different atomic *F* curves given in the literature were tried, the set finally adopted being reproduced in Fig. 4. F_K is that calculated by West[13] for use in the analysis of KH_2PO_4, using a temperature factor found for KCl. We have extrapolated the curve to $F_K=18$ (the number of electrons in the K^+ ion) at $\sin\theta=0$. F_0 is from the corresponding curve given by West, except for a slight increase below $\sin\theta=0.1$ in order to make $F_O=9$ at $\sin\theta=0$. This amounts to an assumption that one electron of each pair shared between *S* and *O* atoms contributes to the structure factor of each of the two atoms. Similarly the F_S curve was obtained by assuming F_S to be everywhere slightly greater than F_P (as calculated by West) and to equal 14 at $\sin\theta=0$. It should be noted that although these particular atomic structure factor curves were chosen in preference to others partly because they gave better agreement with the F_{obs} values for the 00.*l* reflections, they were used without further change to obtain $F_{calc.}$ values for many *hk*.0 reflections and gave very satisfactory agreement.

The effect of slightly varying the parameters *z* and *v* independently was also tried, the values finally adopted as most probable being $z=0.23$ and $v=0.43$. The structure factors calculated on this basis are compared with the "observed" values in Table 3.

TABLE 3

Comparison of relative values of F_{obs} and F_{calc} (on the basis of the final structure) for (00.*l*) reflections.

	(00.1)	(00.2)	(00.3)	(00.4)	(00.5)	(00.6)
F_{obs}	3	4	2	10	6	7
F_{calc}	3.2	4.1	2.3	10.0	3.8	5.3

The approximate values for the *z* coordinates of the oxygen atoms are 0.20, 0.23, 0.34, 0.66, 0.77 and 0.80. They are thus in rather widely separated layers, with each layer containing atoms in three separate levels. By analogy with the known structures of silicates, sulfates and other oxygen compounds, one might expect approximate close-packing within each layer, the oxygens in the three different levels being disposed roughly as are those in three adjacent levels in a cubic close-packed structure. This leads to the approximate arrangement shown in Figure 5. The potassium centers might be expected to be between the oxygen layers, in such a way that each is surrounded by six oxygen centers and is as far as possible from other potassium atoms.

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[13] West, *Zeit. Krist.*, **74**, 306. 1930.

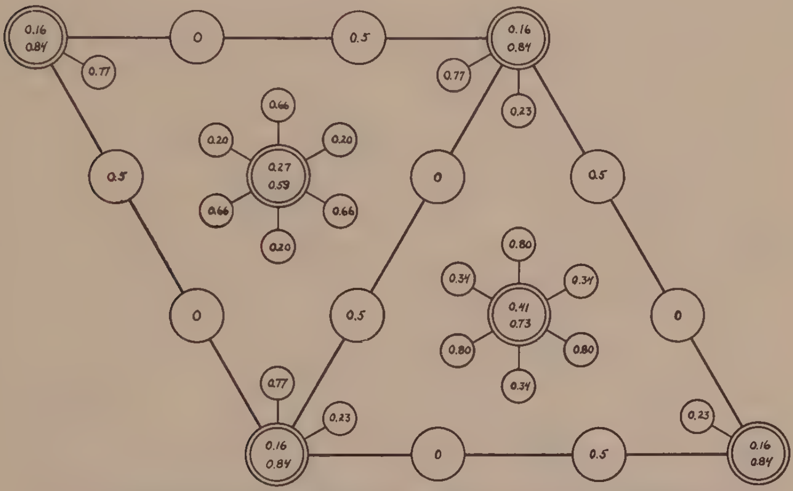

FIG. 5. The approximate arrangement within the unit cell, projected on (00.*l*). Small circles represent oxygen atoms, large single circles potassium atoms, and large double circles pairs of sulfur atoms. The *z* coördinates are shown within the circles.

Calculations on the basis of such an arrangement showed fair agreement with the "observed" (*hk*.0) structure factors, but not good enough. Keeping the distance of each oxygen from the nearest S_2 axis equal to about 1.45–1.65Å and assuming all the K-O distances to be approximately equal to each other, however, resulted in parameters (obtained by a combined graphical and analytical method) which gave much better agreement with experiment. Our "most probable" values, obtained in this way, are listed in Table 4 and a comparison of the F_{obs} and F_{calc} values for the *hk*.0 reflections appearing on one of our films is given in Table 5. Con-

TABLE 4

Parameter values for $K_2S_2O_6$.

S in (c) with $u_s = .16_0$ K in (e) with $u_k = .30_0$
(d) with $u_s' = .59_0$ (f) with $u_k' = .62_5$
(d) with $u_s'' = .27_0$

O in (g) with $x, y, z = .16_5, .11_0, .23_0$
(g) with $x'y'z' = .61_5, .17_0, .34_0$
(g) with $x''y''z'' = .50_5, .21_0, .80_0$

TABLE 5

Indices of (*hk*.0) reflections from a crystal oscillated about the a' axis, with F_{obs} (above the indices) and F_{calc} (below the indices).

						13	
						(07.0)	
			14		52	8	
			($\bar{1}$5.0)		(06.0)		
		39	6	11	55	17	
		($\bar{1}$4.0)		(05.0)		(16.0)	
	21	34	0	12	22	26	
	($\bar{1}$3.0)		(04.0)		(15.0)		
85	19	72	3	14	26	34	
($\bar{1}$2.0)		(03.0)		(14.0)		(25.0)	
75	9	92	30	6	15	29	
	(02.0)		(13.0)		(24.0)		
0	10	13	34	28	12	0	
(01.0)		(12.0)		(23.0)		(34.0)	
3	54	19	9	19	37	7	26
	(11.0)		(22.0)		(33.0)		(44.0)
0	75	13	9	28	67	0	25
(10.0)		(21.0)		(32.0)		(43.0)	
3	9	19	30	19	15	7	
	(20.0)		(31.0)		(42.0)		
85	10	72	34	14	12	34	
(2$\bar{1}$.0)		(30.0)		(41.0)		(52.0)	
75	21	92	0	6	22	29	
	(3$\bar{1}$.0)		(40.0)		(51.0)		
26	19	39	3	11	26	17	
(3$\bar{2}$.0)		(4$\bar{1}$.0)		(50.0)		(61.0)	
19	17	34	14	12	52	26	
	(4$\bar{2}$.0)		(5$\bar{1}$.0)		(60.0)		
56	9	34	6	22	55	13	
(4$\bar{3}$.0)		(5$\bar{2}$.0)		(6$\bar{1}$.0)		(70.0)	
34	43	19	22	26	17	8	
	(5$\bar{3}$.0)		(6$\bar{2}$.0)		(71.0)		
	19	68	12	42	26	19	
		(6$\bar{3}$.0)		(7$\bar{2}$.0)		(8$\bar{1}$.0)	
		67		29		20	

sidering the facts that the intensities were only roughly determined, that no correction for absorption was made and that the atomic structure factors are not accurately known, the agreement is remarkably good. We therefore believe these parameter values to be not far from the correct ones.

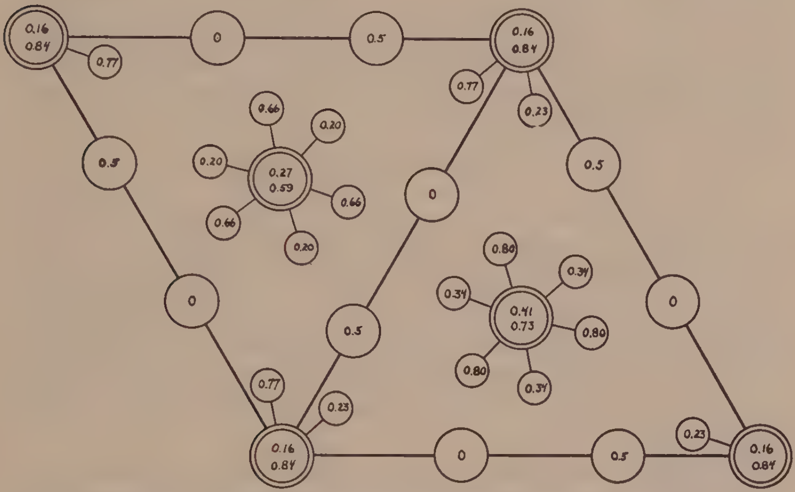

FIG. 6. The same as Figure 5, but with more exact parameter values (Table 4).

DISCUSSION OF THE STRUCTURE

The structure so obtained is diagramatically represented in Fig. 6. It may be considered as an assemblage of K^+ and $S_2O_6^{--}$ ions, the latter having the structure predicted by the Lewis theory, these ions being distributed so as to satisfy as well as possible the following forces: (1) the attraction between positive K^+ ions and negative $S_2O_6^{--}$ ions (especially the O atoms in the latter); (2) the mutual repulsion between oxygen atoms, producing an approximately close-packed arrangement of their centers; and (3) the repulsion between like ions, causing each kind, considered separately, also to be distributed in an approximately close-packed manner.

From the parameter values given in Table 4 the following have been calculated:

Each sulfur is tetrahedrally surrounded by one other sulfur at a distance of 2.0_6Å and three oxygens each at a distance of 1.5_0Å.

Each potassium is surrounded by six oxygen atoms at corners of a distorted octahedron, at distances of 2.7_7–2.8_2Å.

Each oxygen is at a distance of 1.5_0Å from one sulfur and 2.7_7–2.8_2Å from two potassium atoms.

These distances are in excellent agreement with corresponding distances in other compounds. The S-S distance in pyrite (FeS_2) has been determined[14] as 2.09Å. The S-O distance in various sulfates has been found[15] to be 1.5–1.6Å. The K-O distance has been given as 2.79–2.81Å in KH_2PO,[16] as 2.72–2.95Å in $KAl(SO_4)_4$,[17] and as 2.73–3.12Å in $KClO_3$.[18] In the K_2SO_4 structure 10 oxygen atoms are found[19] at distances of 2.71–3.15Å from a potassium center, with an eleventh at 3.50Å.

RUBIDIUM DITHIONATE

Sufficient data were also obtained from $Rb_2S_2O_6$ crystals to show that it has the same type of structure as the potassium salt, as would be expected from the crystallographic data.[20] From the distances between layer lines the unit distances a_0 and c_0 were determined as about 10.0Å and 6.3Å respectively, in agreement with the density, which we roughly determined as 3.00 g./cc., and the axial ratio ($a{:}c=1{:}0.6307$) as given by Groth, if there are three "molecules" per unit.

Although we have made no attempt to determine accurately the parameters a comparison (Table 6) of the observed intensities with values of F^2 calculated on the assumption that the parameters are the same as for $K_2S_2O_6$ (Table 4) shows quite good agreement. We therefore conclude that the true parameters are not very different in the two cases.

In these calculations we used an atomic F curve for Rb obtained from the Cs curve calculated from Thomas' model[21] by the method given by Bragg and West.[22]

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[14] Ewald and Friedrich, *Ann. Physik*, **44**, 1183, 1914.

[15] James and Wood, *Proc. Roy. Soc.*, **A109**, 598, 1925.
Bradley, *Phil. Mag.*, **49**, 1225, 1925.
Dickson and Binks, *Phil. Mag.*, **2**, 114, 1926.
Taylor and Boyer, *Mem. Proc. Manchester, Lit. Phil. Soc.*, **72**, 125, 1928.
Ogg, *Phil. Mag.*, **5**, 354, 1928.
Vegard and Maurstad, *Zeit. Krist.*, **69**, 519, 1929.

[16] West, *Ref. 13.*

[17] Vegard and Maurstad, *Ref. 15.*

[18] Zachariasen, *Zeit. Krist.*, **71**, 501, 1929.

[19] Ogg, *Ref. 15.*

[20] *Ref. 3.*

[21] Thomas, *Proc. Cambridge Phil. Soc.*, **23**, 542, 1927.

[22] W. L. Bragg and J. West, *Zeit. Krist.*, **69**, 118, 1928.

We have also made a study of the structure of $CaS_2O_6 \cdot 4H_2O$, the results of which will be published in another communication.

SUMMARY

1. The unit cell of $K_2S_2O_6$ has the dimensions $a_0 = 9.82$Å and $c_0 = 6.36$Å.

2. The space-group is D_3^2.

3. The distribution of atoms within the unit is as given in Tables 2 and 4 and pictured in Fig. 7.

4. $Rb_2S_2O_6$ has the same type of structure, with $a_0 = 10.0$Å and $c_0 = 6.3$Å and with approximately the same values for the 14 parameters defining the atomic positions.

TABLE 7

Comparison of observed intensities with F^2_{calc} for $hk.0$ and $00.l$ reflections from $Rb_2S_2O_6$.

Intensity scale: s = strong, ms = medium strong, m = medium, mf = medium faint, f = faint, 0 = no observed reflection, although crystal was in the proper position for reflection. A blank indicates no reflection observed, but crystal may not have been in the proper position for reflection.

hk.l	Observed Intensity			Relative F^2_{calc}
	Film 1	Film 2	Film 3	
	Left			
10.0	0	0	0	0
20.0	0	0	0	1
30.0	s	s	s	100
40.0	0	0	0	0
50.0	f	0	0	4
60.0	m	ms	m	33
70.0	0	0	0	3
	Right			
11.0	0	0	0	13
22.0	0	f	0	7
33.0	ms	ms	m	49
44.0	0	0	0	0
21.0	mf	m		5
31.0	m	ms		13
41.0		0		4
51.0		m	mf	13
32.0	mf	m	f	5
42.0	mf	m	mf	12
52.0	f			1
43.0	f			3

hk.l	Intensity Film 4	Relative F^2_{calc}
00.1	0	1
00.2	mf	34
00.3	0	1
00.4	m	100
00.5	0	2

PROCEEDINGS OF SOCIETIES

NEW YORK MINERALOGICAL CLUB

A regular meeting of the New York Mineralogical Club was held at the American Museum of Natural History on the evening of April 15, 1931, with an attendance of 65. President Allen was in the chair.

The meeting was called to order at 8:15 P.M. The Secretary not being on hand, the reading of the minutes and communications were deferred for the moment. The President appointed Dr. Horace R. Blank to act as Secretary *pro tem.*, and then proceeded with the annual election of officers. As President Allen had been nominated for reelection, Mr. Ernest A. Maynard was elected President *pro tem.*

The nominations of the Nominating Committee were read and additional nominations from the floor called for. There were no additional nominations and the election of the following was made unanimous:

Officers of the Club for the present year: President, Frederick I. Allen; first vice-president, George E. Ashby; second vice-president, Horace R. Blank; secretary, Daniel T. O'Connell; treasurer, Gilman S. Stanton.

President Allen resumed the chair. Mr. Morton meanwhile had arrived and relieved Dr. Blank as Secretary. The regular order of business was then resumed. The minutes of the February and March meetings were read and approved. The committee on Membership reported favorably on the names of Mr. John H. Fisher, Mr. W. R. Schneck, Miss Jane Kesler, Mr. H. Alban Anderson, and Miss Lillian Frazer, who were proposed for membership at the March meeting. On motion they were elected to membership. Mr. H. R. Lee, 31 Midland Avenue, White Plains, N. Y., was proposed for membership by Mr. Stanton; Mr. John C. Boyle, Children's Museum, Brooklyn, N. Y., was proposed by Mr. Hoadley.

Communications were read by the Secretary from the following:

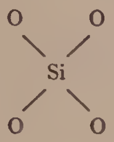
The Roerich Museum of New York, describing the work of Urusvati, their newly organized Himalayan Research Institute and offering joint cooperation in any field of research in their field of interest to the club. They also requested an exchange of publications.

Professor R. J. Colony of Columbia University, expressing regret at being unable to give the lecture scheduled for the February meeting owing to being called away suddenly to report on the effects of an earthquake in Oaxaca, Mexico. He offered to give the lecture announced at any open date in the future.

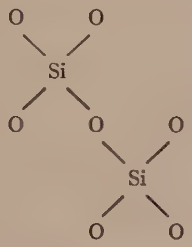
Mineral Department of the British Museum (Natural History), London, England, requesting certain copies of the Club's *Bulletin* which are needed to complete the set on file in their library.

Science Service, Washington, D. C., thanking the club for its offer of cooperation and the copy of Mr. Manchester's book which was forwarded to the Editor of *Science Service* in accordance with the deliberations of the Club at the February meeting, and promising to publish a review of the book in their weekly magazine, the *Science News Letter.*

The newly elected Secretary was then invited to take his post and President Allen introduced the speaker of the evening, Dr. Ralph W. G. Wyckoff, who addressed the club on *The Crystal Structure of the Silicates*. Dr. Wyckoff outlined recent work, due mainly to Professor W. L. Bragg and his students at Manchester University, England, which has been concerned with the difficult task of unraveling the structure of the silicates through the use of *x*-ray diffraction effects. As a result of their study, the silicates no longer need be considered as the salts of a hypothetical series of acids based on analogy with the sulphur and phosphorus acids. It has been found that in every silicate, the silicon atom is at the center of a tetrahedron of oxygen atoms.



These tetrahedrons of SiO_4 have the property of linking up together with other SiO_4 groups resulting in such combinations as Si_2O_7 and Si_3O_{10}, extending on in this manner indefinitely.



These studies have led to the following principles:

1. The existence of a universal SiO_4 group of atoms which are found in all the silicates.
2. The property of these SiO_4 groups to attach themselves together through having one oxygen atom in common.

The structure of the silicate minerals may be classified under four headings:

(A) Discrete silicate groups; (B) Chain structures of linked silicate groups; (C) Sheet structures of linked silicate groups; (D) Network structures of linked silicate groups.

The discrete silicate groups include "Island SiO_4 groups," and more or less complex silicate groups. If these SiO_4 groups do not form islands in crystals they will form *chain structures* and by linking up, double chains and even sheets, may be developed giving *sheet structure*. Sheets linked together would give a 3-dimensional or solid structure of Si and O atoms linked together giving a *network structure*.

The oxygen atoms are relatively very big and the silicon atoms small. The latter fit into the spaces between the oxygen atoms. Thus is explained the enormous stability of the SiO_4 group because of this extra close packing of atoms.

Dr. Wyckoff, by means of lantern slides, showed the atomic structure of a number of the silicate minerals, based on the *x*-ray investigations. Examples of discrete silicate groups mentioned include zircon, olivine, sillimanite, andalusite, cyanite and titanite. In the case of titanite, $CaTiSiO_5$, it was formerly believed that Si and Ti were interchangeable. But the *x*-ray patterns show that this is not the case, for

Si forms a group with 4 oxygen atoms and Ti forms a group with 6 oxygen atoms. In substitutions of one element for another it was found that valence was not the determining factor but rather the size of the atoms. Calcium and sodium atoms substitute for each other readily because their atoms are about the same size.

Examples of chain structures are diopside, and enstatite, which has a chain structure similar to diopside but one in which the chains are in pairs. This doubled chain structure is also found in the amphibole group including tremolite. All the varieties of asbestos show the same structure of chain and paired chain structure including chrysotile. It is thus seen that the chain structure is likely to give a fibrous character to the mineral.

The chain structure and reflected chain structure may give rise by repetition to a sheet structure. The sheet structure gives a micaceous cleavage as in muscovite. This structure is also indicated by preliminary results to be present in talc and the brittle micas also.

Researches on the network structure have not been carried very far as yet but it has been found to be the structure of sodalite, analcite and the zeolites and probably also the feldspars and nephelite.

Dr. Wyckoff completed his lecture at 9:27 P.M. In the questioning that followed, the following points were brought out:

1. The crystal structure as indicated by the *x*-rays is controlled by the chemical composition of the mineral and is not affected by its pseudomorphic structure.
2. When a bivalent element is replaced by a univalent element in a network structure, the replacement is not dependent on the spaces between the building units, necessarily, because the spaces between the units may be expanded.

In closing the discussion, President Allen called attention to Prof. F. W. Clarke's study of the silicates based on valency, "The Constitution of the Silicates," and pointed out that in accordance with the information given to the Club this evening, valency no longer appears to be the dominant factor in the structure of the silicates but rather the size of the atoms.

A rising vote of thanks was voted Dr. Wyckoff for his excellent presentation of this exceedingly important subject.

The meeting adjourned at 9:46 P.M.

DANIEL T. O'CONNELL, *Secretary*

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